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IS 10895 (1984): Copper nitrate [CHD 1: Inorganic Chemicals]



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IS : 10895 - 1984

Indian Standard
SPECIFICATION FOR
COPPER NITRATE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR COPPER NITRATE

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Indian Standard

SPECIFICATION FOR COPPER NITRATE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 19 March 1984, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Copper nitrate, popularly known as cupric nitrate, is an important salt of copper and finds use in light-sensitive reproductive papers, as ceramic colour, as mordant and oxidant in textile dyeing and printing, as reagent for burnishing iron, for giving a black 'antique' finish to copper, for colouring zinc brown, in nickel plating baths, in aluminium brighteners, in wood preservatives, in fungicides and herbicides, in pyrotechnic compositions, as catalyst component in solid rocket fuel, as nitrating agent for aromatic organosilicon compounds and as catalyst for organic reactions.

0.3 Copper nitrate is of two types: $\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$. The first one being unstable above 26.4°C , converting to the $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$. This specification is, therefore, for the stable trihydrate form.

0.4 Copper nitrate is prepared from copper scrap by reaction with nitric acid and crystallizing from the solution. For various uses different purity standards may be necessary. But there is no available standard on this item, except as a reagent grade chemical for use in laboratory for analysis. This standard is intended to be a step towards establishing a workable standard for use by industries and consumers. Any modification or improvement can be incorporated later. Initially it is intended to cover two grades, technical grade for general use and pure grade for specified uses where impurities cannot be tolerated.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for copper nitrate.

2. GRADES

2.1 There shall be two grades of the material, namely,

- a) *Technical Grade* — suitable for all other uses where pure grade is not required, and
- b) *Pure Grade* — suitable for electroplating, catalyst for rocket fuels, catalyst for organic reaction, and analytical applications.

3. REQUIREMENTS

3.1 **Description** — The material shall be in the form of blue deliquescent rhombic crystals or plates having the formula $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$. The material shall be free from visible contamination from dust or black particles and other extraneous adulterants and impurities.

3.2 The material shall also comply with the requirements specified in Table 1 when tested according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR COPPER NITRATE

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Technical Grade	Pure Grade	
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass, <i>Min</i>	98.0	99.0	A-2
ii)	Matter insoluble in acid, percent by mass, <i>Max</i>	0.5	0.01	A-3
iii)	Chloride (as Cl), percent by mass, <i>Max</i>	—	0.002	A-4
iv)	Sulphate (as SO_4), percent by mass, <i>Max</i>	—	0.01	A-5
v)	Substances not precipitated by hydrogen sulphide (as sulphates), percent by mass, <i>Max</i>	—	0.05	A-6
vi)	Lead (as Pb), percent by mass, <i>Max</i>	0.2	0.005	A-8
vii)	Iron (as Fe), percent by mass, <i>Max</i>	0.2	0.005	A-9
viii)	Ammonium sulphide metals other than iron (as Ni), percent by mass, <i>Max</i>	—	0.01	A-10

4. PACKING AND MARKING

4.1 Both technical and pure grade materials shall be packed in polyethylene lined hessian/HDP bags taking care to see that they are properly closed/sealed to avoid contact with air and water.

4.2 Marking — The packages shall be marked legibly and indelibly with the following information:

- a) Name and grade of material;
- b) Mass of the material;
- c) Lot or batch number in code or otherwise; and
- d) Name of the manufacturer and his recognized trade-mark, if any.

4.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples from a lot, number of tests and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR COPPER NITRATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2. ASSAY

A-2.0 Two methods are prescribed for determining copper, namely, volumetric method and electrolytic method. The electrolytic method shall be regarded as the referee method.

*Specification for water for general laboratory use (*second revision*).

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A-2.1 Volumetric Method

A-2.1.0 Outline of the Method — Copper is determined with the addition of potassium iodide and titrating the liberated iodine against standard sodium thiosulphate solution.

A-2.1.1 Reagents

A-2.1.1.1 Concentrated hydrochloric acid — see IS : 265-1976*.

A-2.1.1.2 Glacial acetic acid

A-2.1.1.3 Potassium iodide — solid.

A-2.1.1.4 Standard sodium thiosulphate — 0.1 N

A-2.1.2 Procedure — Weigh accurately about 0.7 g in a 250 ml beaker. Dissolve in 5 ml of water, add 5 ml of hydrochloric acid and evaporate to dryness. Cool, dissolve the residue with 5 ml of water and re-evaporate with 5 ml of hydrochloric acid to dryness on the steam bath. Dissolve the residue in water, transfer the solution to a glass-stoppered 250 ml conical flask and dilute with water to 75 ml. Add 2 ml of glacial acetic acid and 3 g of potassium iodide and then titrate the liberated iodine with 0.1 N sodium thiosulphate, shake and continue the titration until the blue colour disappears.

A-2.1.3 Calculation

Copper nitrate [as $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$],

$$\text{percent by mass} = 24.16 \times \frac{VN}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution,

N = normality of standard thiosulphate solution, and

M = mass in g of the sample taken for the test.

A-2.2 Electrolytic Method — Proceed as per the method given in A-2.2 of IS : 261-1982†.

A-3. MATTER INSOLUBLE IN ACID

A-3.1 Reagents

A-3.1.1 Dilute Nitric Acid — 1:200 (v/v).

*Specification for hydrochloric acid (*second revision*).

†Specification for copper sulphate (*second revision*).

A-3.2 Procedure — Weigh accurately about 10 g of the material and dissolve in 100 ml of dilute nitric acid. Heat to boil, cool to room temperature. Filter through tared sintered glass crucible. Wash the residue with hot water till precipitate is free from acid. Dry the crucible at 105 to 110°C and weigh to constant mass.

A-3.3 Calculation

$$\text{Matter insolubles in acid, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-4. CHLORIDES

A-4.1 Reagents

A-4.1.1 Dilute Nitric Acid — 30 percent (v/v).

A-4.1.2 Silver Nitrate Solution — 0.1 N.

A-4.1.3 Standard Chloride Solution — Dissolve 1.6484 g of pure dry sodium chloride in water and dilute to 1 litre. Dilute 10 ml of this solution to one litre so that one ml contains 0.01 mg of chloride (as Cl).

A-4.2 Apparatus

A-4.2.1 Nessler Cylinders — 20 ml capacity.

A-4.3 Procedure — Weigh 1 g of the material in a 250-ml beaker and dissolve in 20 ml of water, add 1 ml of nitric acid and divide them into two equal portions in different cylinders. To one portion add 1 ml of silver nitrate solution, bring to a boil, let stand for 10 minutes. Cool, filter until clear and use this solution for the control test. To the other portion in the second cylinder add 1 ml of silver nitrate solution. If a turbidity is produced, it is not greater than is produced by adding 1 ml of standard chloride solution to the control and adjusting both to the same volume.

A-5. SULPHATES

A-5.1 Reagents

A-5.1.1 Dilute Hydrochloric Acid — 1:2 (v/v).

A-5.1.2 Dilute Hydrochloric Acid — 1:99 (v/v).

A-5.1.3 Barium Chloride Solution — 0.5 M (122.1 g of barium chloride dissolved in water and made up to 1 000 ml).

A-5.2 Procedure — Dissolve 5 g of the material in 15 ml of hot dilute hydrochloric acid (1:2) and evaporate to dryness on the steam bath. Dissolve the residue in about 15 ml of hot dilute hydrochloric acid (1:2) and again evaporate to dryness. Dissolve the residue in 100 ml of dilute hydrochloric acid (1:99) and filter. Heat to boiling, add 5 ml of barium chloride solution, digest in a covered beaker on the steam bath for 2 hours and allow to stand overnight.

A-5.2.1 If a precipitate is formed, filter, wash thoroughly and ignite. The mass of the precipitate should not be more than 0.0012 g greater than the mass obtained in the blank.

A-6. SUBSTANCES NOT PRECIPITATED BY HYDROGEN SULPHIDE

A-6.1 Reagents

A-6.1.1 *Hydrogen Sulphide* — gas.

A-6.1.2 *Dilute Sulphuric Acid* — 1:95 (v/v).

A-6.2 Procedure

A-6.2.1 Dissolve 4 g of the material in about 190 ml of dilute sulphuric acid, heat to about 70°C, and pass in hydrogen sulphide until all the copper is precipitated. Dilute to 200 ml and filter. Evaporate about 150 ml of the filtrate to dryness in a tared dish and ignite.

A-6.2.2 The mass of the residue shall not exceed 0.0015 g. Reserve the residue for the preparation of sample solution A (A-7).

A-7. SAMPLE SOLUTION A — To the residue obtained in the test for substances not precipitated by hydrogen sulphide (A-6.2.2), add 3 ml of dilute hydrochloric acid (1:1) and 0.15 ml of nitric acid, cover with a watch glass, and digest on the steam bath for 15 to 20 minutes. Remove the watch glass and evaporate to dryness. Dissolve the residue in 1 ml of hydrochloric acid and dilute to 30 ml (1 ml = 0.1 g).

A-8. LEAD

A-8.1 For Pure Grade

A-8.2 Reagents

A-8.2.1 *Dilute Nitric Acid* — 1:99 (v/v).

A-8.2.2 *Lead* — Foil.

A-8.2.3 *Hydroxylamine Hydrochloride* — Solution.

A-8.2.4 *Dilute Ammonium Hydroxide* — 1 N, approximately.

A-8.2.5 *Ammonium Citrate*

A-8.2.6 Potassium Cyanide Solution

A-8.2.7 Dithizone Solution — 0.01 percent in chloroform. This solution is to be freshly prepared.

A-8.2.8 Dilute Dithizone Solution — 0.001 percent. Dilute 10 ml of the solution (A-8.2.7) to 100 ml with chloroform.

A-8.3 Procedure — In a well ventilated hood dissolve 1.5 g in 15 ml of dilute nitric acid in a glass-stoppered cylinder. Prepare a control containing 0.5 g of sample and 0.01 mg of lead ion (Pb) in 50 ml of dilute nitric acid in a glass-stoppered cylinder. To each solution add 2 ml of hydroxylamine hydrochloride reagent solution for dithizone test. Add ammonium hydroxide dropwise until a permanent precipitate forms, shaking well after the addition to each drop. Add 5 ml of ammonium citrate reagent solution and 25 ml of potassium cyanide reagent solution. Cool in ice water for 15 minutes. To each add 3 ml of dithizone standard reagent solution in chloroform, shake for 2 minutes, and allow the layers to separate. Any pink colour of the chloroform layer from the solution of the sample should not exceed that of the chloroform layer from the control.

A-8.4 For Technical Grade

A-8.4.1 Carry out the test as prescribed in IS : 7017 - 1973*.

A-9. IRON**A-9.1 For Pure Grade****A-9.2 Apparatus**

A-9.2.1 Nessler Cylinders — 50 ml capacity.

A-9.3 Reagents

A-9.3.1 Concentrated Hydrochloric Acid — see IS : 265 - 1976†.

A-9.3.2 Ammonium Persulphate — Solid.

A-9.3.3 Butanolic Potassium Thiocyanate Solution — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up 100 ml and shake vigorously until the solution is clear.

A-9.3.4 Standard Iron Solution — Dissolve 0.702 g of ferrous ammonium sulphate [Fe(NH₄)₂SO₄.6H₂O] in 10 ml of dilute sulphuric acid (10 percent by volume) and dilute with water to exactly 1 000 ml. Pipette out 10 ml of the solution and dilute with water to make up the volume to exactly 100 ml. One millilitre of this solution contains 0.01 mg of iron (Fe).

*Methods for colorimetric determination of traces of heavy metals by dithizone.

†Specification for hydrochloric acid (second revision).

A-9.4 Procedure — Dissolve 1.0 g of the material in 20 ml of water and transfer to a Nessler cylinder. Add 1 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up the volume to 50 ml. Shake vigorously for 30 seconds and allow the butanol layer to separate. Carry out a control test in the other Nessler cylinder using 5 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the two cylinders after 2 minutes.

A-9.4.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the control test.

A-9.5 For Technical Grade

A-9.6 Reagents

A-9.6.1 Nitric Acid — see IS : 264-1976*.

A-9.6.2 Hydrochloric Acid — see IS : 265-1976†.

A-9.6.3 Ammonia

A-9.6.4 Ammonium Chloride

A-9.7 Procedure — Weigh accurately about 10 g of the material and dissolve it in 10 ml nitric acid and 100 ml of water by heating. Precipitate iron by adding ammonia till the colour of the solution turns deep blue, heat to boil and filter. Wash with dilute solution of ammonia till the washings are colourless. Dissolve the residue in 10 ml of hydrochloric acid and 50 ml of water taken in a beaker. Add 5 g of ammonium chloride and stir to dissolve. Add 20 ml of ammonia, boil, cool, filter. Wash the precipitate with water, dry the residue, ignite, and weigh to constant mass.

A-9.8 Calculation

$$\text{Iron content, percent by mass} = \frac{M_1 \times 70}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-10. AMMONIUM SULPHIDE METALS OTHER THAN IRON

A-10.1 Apparatus

A-10.1.1 pH meter.

*Specification for nitric acid (*second revision*).

†Specification for hydrochloric acid (*second revision*).

A-10.2 Reagents

A-10.2.1 Dilute Ammonium Hydroxide — 15 percent NH_3 (v/v).

A-10.2.2 Dilute Hydrochloric Acid — 1:1 (v/v).

A-10.2.3 Hydrogen Sulphide Water — saturated.

A-10.2.4 Standard Nickel Solution — Dissolve 6.73 g of nickel ammonium sulphate $[\text{NiSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ in water and dilute to 1 000 ml. Dilute 10 ml of the solution to 1 000 ml. One millimetre of the solution contains 0.01 mg of nickel (as Ni).

A-10.3 Procedure — Ammonium sulphide metals other than iron. To 10 ml of Sample Solution A (A-7) add a slight excess of ammonium hydroxide, boil for 1 minute, filter, and wash with a small quantity of hot water. Dilute the filtrate and washings to 25 ml. Adjust the pH of 5 ml of this solution to 7 (using a pH meter) with dilute hydrochloric acid, and dilute to 20 ml. Add 0.10 ml of ammonium hydroxide and 1 ml of hydrogen sulphide water. Any colour should not exceed that produced by 0.02 mg of nickel ion (Ni) in an equal volume of solution containing the quantities of reagents used in the test.

APPENDIX B

(Clause 5.1)

SAMPLING OF COPPER NITRATE**B-1. GENERAL REQUIREMENTS OF SAMPLING**

B-1.0 In drawing, preparing, storing and handling test samples the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material have no action.

B-1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables (see IS : 4905 - 1968*) shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them 1, 2, 3 , up to r and so in a systematic manner, where r is the integral part of N/n . Every r th container thus counted shall be taken so as to constitute the requisite sample size.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

(Clause B-2.2)

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
(N)	(n)
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-3. TEST SAMPLES AND REFEREE SAMPLE**B-3.1 Preparation of Test Samples**

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under 3 and shall not exceed 1 kg.

*Methods for random sampling.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, second for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portions of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under B-1.4. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets or samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (*see* B-3.1.2) and a set of individual samples (*see* B-3.1.3) marked for this purpose. It shall also bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Test for the determination of copper nitrate shall be conducted on each of the individual samples for all the grades.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 For Copper Nitrate — The test results for copper nitrate shall be recorded, and the mean and the range for these test results shall be calculated as follows:

Mean (\bar{X}) = Sum of the test results divided by the number of test results,

Range (R) = The difference between the maximum and the minimum values of the test results.

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The lot shall be declared to have satisfied the requirement for this characteristic if the expression $(\bar{X} - 0.4 R)$ is greater than or equal to the prescribed limits in Table 1.

B-5.2 For Composite Sample — The test results on the composite sample shall meet the corresponding requirements specified in Table 1 for the relevant grade.

B-5.3 The lot shall be declared as conforming to the requirements of the specification if **B-5.1** and **B-5.2** are satisfied.

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SHRI M. VARADARAJAN (*Alternate*)
 SHRI J. T. VORA
 DR ANIL PANDIT (*Alternate*)

Representing

The Millowners' Association, Bombay
 Development Commissioner, Small Scale Industries, New Delhi

Central Mining Research Station (CSIR),
 Dhanbad
 Tamil Nadu Chromates & Chemicals Ltd,
 Madras

Deepak Nitrite Ltd, Vadodara